

# Development and Experimental Investigation of Pigeon Pea Stalk Particle Reinforced Epoxy Composites and their Hybrid Composites for Lightweight Structural Applications

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The pigeon pea (*PP*) stalk is a sustainable lignocellulosic material left by the farmers after harvesting its pulses. The use of agricultural residue in the development of polymer composites is a step towards sustainability. This study focuses on developing and characterizing the mechanical properties (the tensile, flexural, interlaminar shear, compression, impact, and hardness) of less utilized agro-based *PP* stalk particle reinforced epoxy composites and their hybrid composites. In addition, the density, dynamic mechanical analysis, water absorption, and morphology were also investigated for a better understanding of these composites. In comparison to other agro-residue reinforced composites, *PP* stalk particles (up to 20 wt.%) reinforced epoxy composites have demonstrated comparable mechanical, viscoelastic, and water absorption characteristics. Jute/*PP*/epoxy and glass/*PP*/epoxy hybrid composites outperformed *PP*/epoxy composites in mechanical, dynamic, and water absorption characteristics. The ranking of the composites based on the characterization was done using the TOPSIS method, and glass/*PP*/epoxy composite with a 20 wt.% was identified as the best performer among all the composites. The results demonstrated that *PP* stalk particle reinforced composites are a viable alternative to wood and other natural fiber-based composites and could be used in lightweight structural applications such as automotive interiors, furniture, packaging containers, and cascading applications.

**Keywords:** *Agro-residue, mechanical properties, dynamic mechanical analysis, hybrid composites, TOPSIS.*

## 1. Introduction

Due to the new regulations regarding the environmental concern, there is an increasing demand for natural or renewable resources (natural fibers and polymers) to develop eco-friendly and sustainable composites that meet the needs of present and future infrastructure materials in many sectors like civil, automobile, home appliances, packaging, and so on. These materials possess numerous advantages such as biodegradability, reusability, low greenhouse gas emissions, higher specific properties, cost-effectiveness, and non-toxicity compared to synthetic materials. Lignocellulosic materials are derived from purposely grown plants or residues of agriculture. The purposefully grown fibers are expensive when compared to agricultural leftover fibers. Since agricultural plants can accomplish two goals simultaneously, food production is the first concern, followed by composite development, pulp extraction, electricity generation<sup>1</sup>, etc. In India, nearly 611 million tons<sup>2</sup> of agricultural residue are being produced annually, and this surplus material could be used to establish new bio-based companies in the future. The usage of agro-residues in developing composites has several advantages, including ample supply, cost-effective manufacturing, reducing health hazards (open-air dumping and burning), contribution towards farmer empowerment, and many more<sup>3</sup>.

In recent years a multi-fiber composite has been considered to overcome the disadvantages of single fiber composites.

Abaca-jute-glass hybrid composites exhibited the enhancement of mechanical properties and the improvement in the surface finish<sup>4</sup>. Sisal/bagasse hybrid composites proved better mechanical characteristics than bagasse epoxy composites<sup>5</sup>. Adding five wt.% aramid fiber in bagasse/epoxy composites improved tensile, flexural, and impact strengths<sup>6</sup>. An enhancement of flexural and impact properties was also achieved by hybrid composites made of banana and sisal fibers<sup>7</sup>. In another study, olive pomace-filled glass/epoxy composites exhibited excellent mechanical and flexural strengths<sup>8</sup>.

Pigeon pea (*Cajanus cajan*), which belongs to the family of Fabaceae, is the sixth most important legume food crop worldwide. It is grown in nearly 82 countries, and almost 5.4 million hectares<sup>9</sup> are being cultivated each year globally. The pigeon pea (*PP*) plant grows about 3 meters tall in 6-8 months. The stalk contains about 55% cellulose<sup>10</sup>, comparable to other natural fibers such as sugarcane, jute, hemp, etc. The residue-to-crop ratio of *PP* is more than sugarcane, banana, coconut, maize, and wheat crops<sup>11</sup>. A limited number of studies have been observed on *PP* stalk materials compared to other natural fibers. The studies include; the extraction of pulp<sup>12</sup> for paper industries, xylooligosaccharides production<sup>13</sup>, cement-bonded composite boards development<sup>14</sup>, generation of fuel gas<sup>15</sup>, and characterization of *PP*/polypropylene composites<sup>16</sup>. The ample availability of *PP* stalk material has encouraged the authors to investigate its potential use as reinforcement in composite development for lightweight structural applications such as

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the development of automobile interiors, furniture, indoor civil structures, packaging containers, and so on. Various treatments have been reported in the literature to enhance the mechanical properties of plant fibers<sup>17</sup>. As a preliminary step and due to environmental concerns, no chemical treatment has been considered for *PP* stalk material to develop eco-friendly composites.

To the author's knowledge from the literature survey, no work has been considered on the development and characterization of *PP*/epoxy (EP) composites, jute/*PP*/epoxy (EJP), and glass/*PP*/epoxy (EGP) hybrid composites. The main objective of this study was to utilize agro-based *PP* stalk material effectively to develop an eco-friendly, cost-effective, and new class of epoxy-based composites and investigate physical, mechanical, viscoelastic, and water absorption characteristics. EP composites were produced in three different weight percentages of *PP* stalk particles (15, 20, and 25). Jute and glass bi-woven fabrics (as skin materials) were used to develop hybrid composites to check the compatibility and to notice the enhancement of mechanical and viscoelastic properties.

## 2. Materials and Methods

### 2.1. Materials

The *PP* crop is an abundantly growing food crop for its pulses in almost all countries. The materials used in this study were *PP* stalk particles, bi-woven jute and glass fiber mat, and commercially available epoxy resin. The raw *PP* stalks were collected from farmers after harvesting their pulses. The stalks cannot be used as continuous fiber and must be converted into flakes, particles, or pulp. Therefore, the stalks were cut to around 30 cm length using a sickle and further to a length of 3 cm to 5 cm using a wood saw for further processing. *PP* stalks were soaked in freshwater for about two days to soften them and remove dust and soluble contaminants. These cut pieces were kept in open sunlight for moisture removal for about three days. The additional moisture content was removed using an air circulating oven at 80° C for 24 hours until constant mass. These *PP* stalk cut pieces were crushed into particles using a mechanical pulverizer. From the sewing process, the particles having

less than 2 mm were isolated and considered for developing EP composites, EJP, and EGP hybrid composites.

Bi-woven jute fiber mat (260 GSM and 1.45g/cc) and glass fiber mat (380 GSM and 2.5g/cc) were procured from Gogreen products, Chennai, India, used as a skin ply in the present study. LY 556 epoxy resin (viscosity 10000-12000 mPa-s, epoxide index 5.3-5.45 Eq/kg, and density 1.15-1.2 g/cc) was used as a matrix material owing to its excellent mechanical strength and adhering quality with fiber materials. Both epoxy and hardener (HY951) were supplied by Herenba Instruments and Engineers, Chennai, India.

### 2.2. Methods

#### 2.2.1. Preparation of EP composites, EJP, and EGP hybrid composites

EP composites, EJP, and EGP hybrid composites were developed by the simple hand layup procedure using a 250mm x 250 mm x 5 mm metallic mold followed by a light compression molding technique. The weight fraction of *PP* stalk particles, epoxy, bi-woven jute fiber, and glass fiber for the development of composites is given in Table 1. The EP composites were developed for three compositions, namely EP1, EP2, and EP3. Two polyethylene sheets and wax (releasing agent) were used to prevent composites from sticking to the mold during composite development. The required amount of *PP* stalk particles and matrix (mixture of epoxy and hardener with a 10:1 ratio) as per the different weight fractions were mixed thoroughly for about 15 minutes and poured into the mold. A mild ramming process was used to achieve uniform settlement of *PP* stalk particles and matrix materials in the mold. Using rollers, the entrapped air bubbles were removed. In the next step, the entire mold assembly was shifted to a hydraulic press, where a pressure of 100 bar was applied at room temperature and left to cure for 24 hours. After curing, the EP composite was demoulded and trimmed. In EJP hybrid composites preparation, a layer of jute fiber mat impregnated with matrix was placed at the bottom side of the mold, and entrapped air bubbles were eliminated with the help of rollers. And then, the mixture of *PP* stalk particles and epoxy was poured into the mold, followed by adding a second layer of jute/epoxy at the top. After shifting the mold to a hydraulic press, a pressure of

**Table 1.** Specimen designation with the composition.

Sl. No.	Composite Specimen Designation	Fiber and matrix weight fraction in %					Thickness in mm
		Core		Skin			
		PP	Epoxy	Jute (2 layers)	Glass (2 layers)	Epoxy	
1	EP0	-	100	-	-	-	5.0
2	EP1	15	85	-	-	-	5.0
3	EP2	20	80	-	-	-	5.0
4	EP3	25	75	-	-	-	5.0
5	EJP1	15	85	40	-	60	5.0
6	EJP2	20	80	40	-	60	5.0
7	EJP3	25	75	40	-	60	5.0
8	EGP1	15	85	-	40	60	5.0
9	EGP2	20	80	-	40	60	5.0
10	EGP3	25	75	-	40	60	5.0

100 bar was applied at room temperature and allowed to cure for about 24 hours. After curing, the hybrid composite was demoulded and trimmed. The same procedure was repeated for the development of EGP hybrid composites. Pure epoxy lamina was also developed for comparison purposes. Figure 1 depicts the preparation of *PP* stalk particles and the development of EP composites, EJP, and EGP hybrid composites.

### 2.2.2. Specimen preparation

Composite specimens were cut using an electrical plate cutting machine as per different ASTM standards. To ensure consistency in results, five specimens for each test were prepared. Before testing, all specimens were dried in an oven at 50 °C for 12 hours and maintained in vacuum bags.

### 2.2.3. Test methods

The theoretical density ( $\rho_T$ ) of composites specimens was evaluated from Equation 1 (rule of mixture) and the actual density ( $\rho_A$ ) was determined by METTLER TOLEDO ME204 analytical balance equipment (Archimedes principle)

as per ASTM D792-08. The void volume content percentage ( $V_v$ ) of all composites is calculated from Equation 2.

$$\rho_T = \frac{1}{\left(\frac{W_{PP}}{\rho_{PP}}\right) + \left(\frac{W_M}{\rho_M}\right) + \left(\frac{W_E}{\rho_E}\right)} \quad (1)$$

$$V_v = \left(\frac{\rho_T - \rho_A}{\rho_T}\right) * 100 \quad (2)$$

Here,  $W$  and  $\rho$  represent weight fraction and density, whereas suffix  $PP$ ,  $M$ , and  $E$  indicate pigeon pea, jute (or glass) biwoven mat, and epoxy, respectively.

The tensile, flexural, interlaminar shear, and compression tests were conducted at room temperature around 25 °C and relative humidity of 60% using the Tinius Olsen 50 KS Universal Testing Machine. Tensile testing was carried out on “dog bone” shaped specimens (165×13×5 mm<sup>3</sup>) to evaluate tensile strength, young’s modulus, and elongation at a speed of 3 mm per minute as per ASTM D 638-14. The flexural test was carried out at a crosshead speed of 3 mm per minute on rectangular specimens (80×20×5 mm<sup>3</sup>) to determine the



**Figure 1.** Preparation of *PP* stalk particles and the development of EP composites and their hybrid composites.

flexural strength and flexural modulus. Both the interlaminar shear strength (specimen size,  $20 \times 10 \times 5 \text{ mm}^3$ ) (ILSS) and compressive strength ( $\sigma_c$ ) (specimen size,  $12.7 \times 10 \times 5 \text{ mm}^3$ ) tests were carried out at a crosshead speed of 1 mm per minute as per ASTM D 2344 and ASTM D 695-15 respectively using Tinius Olsen 50 KS universal testing machine (UTM). The load and displacement data were recorded using a data acquisition system. The ILSS and maximum compressive strength were calculated from Equations 3 and 4.

$$ILSS = 0.75 * \frac{P_{Max}}{b * t} \quad (3)$$

$$\sigma_c = \frac{P_{Max}}{b * t} \quad (4)$$

Here, 'P' is the maximum load carried by the specimen, and 'b' and 't' are related to the composite specimen's breadth and thickness, respectively. Energy absorbed by each composite specimen ( $65 \times 12.7 \times 5 \text{ mm}^3$ ) was noted down using a computerized impact tester (release angle of  $150^\circ$ ) as per ASTM D256. Shore D hardness testing was carried out with a tester's indenter diameter of 1.25 mm and a conical angle of  $30^\circ$  as per ASTM D 2240. The DMA test was carried out using DMA Q 800, V 7.4 BUILD 126, dual cantilever, and multi-frequency strain module equipment as per ASTM D 5418 on composite specimens having dimensions  $55 \times 10 \times 5 \text{ mm}^3$ . The temperature from  $25^\circ\text{C}$  to  $180^\circ\text{C}$  was considered at a variable frequency of 1 Hz to 10 Hz, strain of 0.0345%, and heating rate of  $5^\circ\text{C}/\text{min}$ . In this study, a variable frequency was considered to understand the characteristics of a composite at multiple frequencies. A water absorption test was performed on composite specimens with dimensions  $76 \times 25 \times 5 \text{ mm}^3$  as per ASTM D 570-98 for up to 30 days using distilled water. The weight gain of each composite specimen was recorded every day for the first five days, then every five days for the next 25 days. The percentage of water absorption is determined using Equation 5.

$$Water\ absorption(\%) = \left( \frac{m_{wet} - m_{dry}}{m_{dry}} \right) * 100 \quad (5)$$

Here,  $m_{wet}$  and  $m_{dry}$  are the wet and dry mass of the composite specimens after and before water immersion, respectively. Zeiss (EVO 18) scanning electron microscope images were used to observe the uniform dispersion of PP stalk particles in the matrix and understand the composites' failure mechanism (tensile and flexural fractured specimens). Due to conductivity issues, a thin layer of gold was applied on fracture surfaces (sputtering) before conducting the experiments.

### 3. Results & Discussion

#### 3.1. Density and void assessment

The PP stalk fiber has a density of  $1.7389 \text{ g/cc}^{10}$ . This is 16.6% more than natural jute fiber and 43.7% less than synthetic glass fiber (from suppliers). Also, PP stalk fiber is marginally denser than its pod fiber<sup>18</sup>. Table 2 depicts the theoretical, actual, and void content results of EP composites, EJP, and EGP hybrid composites. The density of PP stalk particles is more than epoxy. Therefore, an increase in fiber content in the matrix increases the theoretical density. But the

**Table 2.** Theoretical density, actual density, & void content of EP composites, EJP, and EGP hybrid composites.

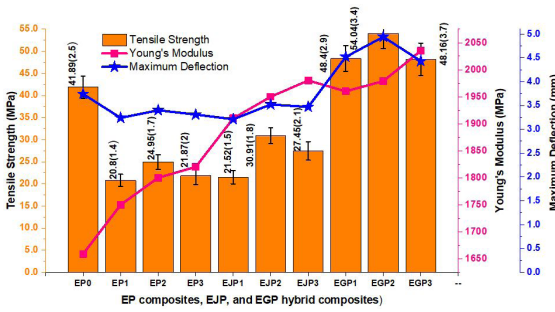
Composite specimens	Theoretical density (g/cc)	Actual density (g/cc)	Void (%)
EP0	1.170	1.131 (0.002)	3.3
EP1	1.230	1.182 (0.005)	3.9
EP2	1.252	1.191 (0.008)	4.9
EP3	1.274	1.174 (0.009)	7.8
EJP1	1.223	1.193 (0.003)	2.5
EJP2	1.252	1.199 (0.006)	4.2
EJP3	1.274	1.191 (0.008)	6.5
EGP1	1.293	1.262 (0.003)	2.4
EGP2	1.315	1.267 (0.005)	3.7
EGP3	1.338	1.254 (0.006)	6.3

experimental density depends on both fiber and void percentage in the composites. The EP3 composite has a maximum void content than EP1 and EP2 composites. Similarly, the hybrid composites (EJP3 and EGP3) showed higher void content than their counterparts. The presence of void content in any composite significantly influences the mechanical properties. In EP3, EJP3, and EGP3 composites, more fiber content reduces a sufficient cross-linking between fiber and matrix, resulting in lower mechanical properties. In general, voids are considered imperfections in any composites. However, the hand layup procedure cannot eliminate the complete void content. The void content in hybrid composites (up to 20 wt.% PP stalk particles) is significantly reduced compared to EP composites. The low void percentage in jute and glass hybrid laminates indicates a better interlaminar adhesion. From the experiments, all EP composites and hybrid composites (EJP and EGP) up to 20 wt.% PP stalk particles exhibited less than 5% void content. Composites having less than 5% void content are allowed for general purpose applications (except aerospace applications)<sup>19</sup>.

#### 3.2. Tensile properties

The EP2 composite showed a maximum tensile strength when compared to its counterparts. The tensile strength in EP composites decreased with the addition of PP stalk particles to the matrix when compared to a pristine epoxy specimen, as depicted in Figure 2. However, the addition of PP stalk particles to epoxy matrix promotes eco-friendly and economical engineering products.

PP/epoxy composites were significantly stronger at 20 wt.% PP particles when compared to their counterparts owing to the effective load transfer between the PP stalk particles and epoxy matrix. Maximum tensile strength of 24.95 MPa was noticed for EP2 composite, which is superior to 30 wt.% banana epoxy composites<sup>20</sup> and almost close to sugarcane bagasse epoxy composites<sup>21</sup>. The average values of tensile strength and standard deviation (given in bracket) have been calculated for each specimen. The hybrid composites EJP2 and EGP2 revealed remarkable improvement in tensile strength. Nearly 23.88% and 147% improvement in tensile strength was observed in EJP2 and EGP2 composites compared to EP2 composite. Here, the jute fiber and glass fiber plies bear most of the external load. In addition, these



**Figure 2.** Tensile characteristics of Epoxy, EP composites, and their hybrid composites.

fibers reduce the stress concentration in the composites resulting in the uniform distribution of stress over the entire composite without inducing intralaminar material failure (minimizing fracture formation). Glass and jute fibers used in this study are continuous, stiff, and strong compared to discontinuous Pigeon pea stalk particles and thus decrease the degree of stress concentration (no sudden change in geometry of fibers) and reduce the interlaminar material failure. The maximum young's modulus noticed in EP composites was 1820 MPa (EP3) which is 9.7% more than pristine epoxy. This was further enhanced in EJP and EGP hybrid composites by 8.8% and 11.8%, respectively. Jute and glass fibers have exceptional mechanical characteristics, take a large percentage of the tensile load during testing, and serve as a protective cover for the base composites (EP composites). The maximum deflection of 3.4 mm was observed in EP2 composite, which is 9.1% less than epoxy. The EJP2 and EGP2 hybrid composites showed 3.5% and 45.3% more elongation than the EP2 composite. Finally, the findings showed that hybrid composites are better performers than EP composites.

### 3.3. Interlaminar shear strength (ILSS)

Table 3 depicts the average value (five specimens) of maximum shear load, shear deflection, and ILSS of each composite. The standard deviation of each value is given in brackets. Randomly distributed *PP* stalk particles in the epoxy matrix reduced the ILSS of EP composites owing to rapid shearing of particles in composites (like tensile strength characteristics). However, the inclusion of jute and glass fibers in the EP composites enhanced the ILSS. The increase in void content in the composite significantly affects the composite's ILSS. As per the standards, specimens with a smaller span-to-depth ratio (less than 6) will improve shear strength accuracy. The present work considered the span-to-length ratio of 4 for all the composite specimens. The failure type, position, and characteristics of the load-displacement curve were observed until the specimen was completely failed. Specimen failure location and mode were identified by the deviation of the load-displacement curve from the typical load-displacement curve. All EP composites, EJP, and EGP hybrid composites were experienced an interlaminar shear failure among all three usual failure modes (interlaminar shear, flexure, and inelastic deformation) of shear. EP composites and EJP composites showed lower deformation, but EGP

**Table 3.** ILSS characteristics of EP composites, EJP, and EGP hybrid composites.

Composite specimens	Shear load (N)	Shear deflection (mm)	ILSS (MPa)
EP0	1800 (20)	0.832 (0.002)	27.0 (0.30)
EP1	1245 (22)	0.864 (0.003)	18.7 (0.33)
EP2	1100 (26)	0.894 (0.003)	16.5 (0.39)
EP3	1030 (31)	0.832 (0.005)	15.5 (0.46)
EJP1	1488 (21)	1.012 (0.002)	22.3 (0.32)
EJP2	1250 (24)	1.245 (0.004)	18.8 (0.36)
EJP3	1220 (28)	1.134 (0.004)	18.3 (0.42)
EGP1	2066 (15)	1.821 (0.001)	31.0 (0.22)
EGP2	1916 (20)	1.924 (0.002)	28.7 (0.30)
EGP3	1728 (32)	1.815 (0.003)	25.9 (0.35)

hybrid composites showed higher deformation during ILSS testing. This indicates that EGP composites have higher flexibility than EP composites. EP1 composite showed a maximum ILSS of 18.7 MPa (based on the short beam strength equation). However, an improvement of 19.3% and 65.77% was noticed in EJP1 and EGP1 hybrid composites, respectively, compared to EP1 composite. This improvement shows that jute and glass fibers have better shear characteristics than *PP* stalk particles.

### 3.4. Flexural properties

Figure 3a shows the average value of flexural strength and flexural modulus of all the composites. The standard deviation for each composite is provided in the brackets. The EP2 composite has a maximum flexural strength (49.3 MPa) compared to its counterparts. This shows that EP2 composite has optimum *PP* stalk particles to transfer the load to the matrix. The higher fiber content reduced the fiber-matrix adhesion, thereby causing the reduction in the flexural strength. Usually, composite specimens experience tensile, compression, and shearing loads during flexural testing. Because of the lower shearing strength of *PP* stalk particles, EP composites showed lesser flexural strengths. However, the hybrid composites showed remarkable improvement in flexural strength. The flexural strength was increased by 20.4% and 127.0% in EJP2 and EGP2 hybrid composites, respectively. Here, the jute and glass fibers enhance the interfacial adhesion and improve the additional site for mechanical interlocking. In addition, jute and glass fibers provide excellent shear resistance to *PP* stalk composites. A similar enhancement of flexural strength has been noticed after the addition of glass fiber to ramie fiber in the study of glass/ramie/epoxy hybrid composite<sup>22</sup>. The *PP* stalk particles in the epoxy matrix positively impacted flexural modulus. The EP3 has demonstrated the highest flexural modulus (4200.25 MPa). This is 36.9% more than pure epoxy lamina. The improvement in flexural modulus has been noticed in hybrid composites in the order of 2.32% and 53.3%, respectively. This could be due to the higher stiffness of jute and glass fibers in the hybrid composites. This concludes that the continuous fibers are more effective

than discontinuous fibers or particles in polymer composites during load transfer<sup>23</sup>.

### 3.5. Compressive strength

PP stalk composites and their hybrid composites both showed an increase in compressive strength with the addition of PP stalk particles up to 20 wt.%. The average value and corresponding standard deviation for each specimen are given in Figure 3b. EP2 exhibited an increase in compressive strength of 6.5% compared with the epoxy matrix. This confirms that the PP stalk particles have excellent compressive load-bearing capabilities. EJP2 and EGP2 hybrid composites exhibited a marginal increase of 8.4% and a significant increase of 23.25% in compressive strength compared to epoxy matrix, respectively. Here, fiber continuity plays a vital role in transmitting compression load from the loading end to the supporting end. Similar behavior has been observed in the study of birch, palm, and eucalyptus fibers with epoxy<sup>24</sup>. Here, 20 wt.% fibers in the matrix have shown an excellent compressive property.

### 3.6. Impact strength

The chemical composition (cellulose, lignin, wax, etc.) of natural fibers plays a vital role in deciding the impact strength of the natural fiber-based composites. Thus, the impact strength of natural composite depends on the amount and type of natural fiber present. As seen in Figure 4a, the increasing trend in impact strength is correlated with the increase in PP stalk particle content. The higher cellulose content and lignin present in the PP stalk particles absorb most of the impact load and thereby cause the improvement in the impact properties of the composites. A similar trend has been observed in the study of areca husk polymer composites<sup>25</sup> up to 40 wt.% fiber. A maximum of 3.97 kJ/m<sup>2</sup> was noticed in the EP3 composite. A 26.2% increase in impact strength was noticed in the EJP3 hybrid composite compared to the EP3 composite. The excellent impact strength (nearly 11.6 times) was observed in the EGP3 hybrid composite compared to the EP3 composite due to the higher toughness (greater energy absorption) and strength of glass fiber.

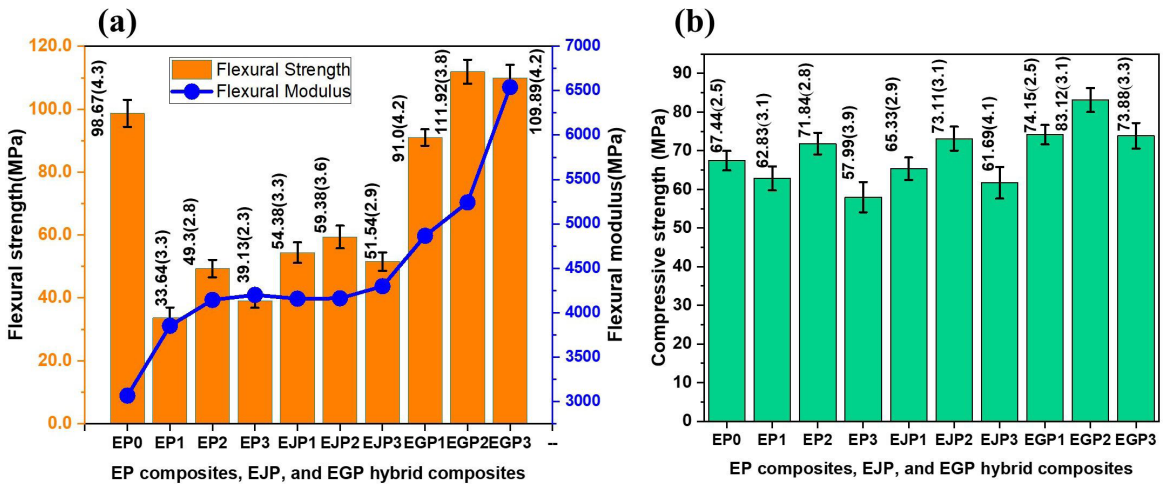


Figure 3. Mechanical characteristics of Epoxy, EP composites, and their hybrid composites: (a) Flexural characteristics, (b) Compressive strength.

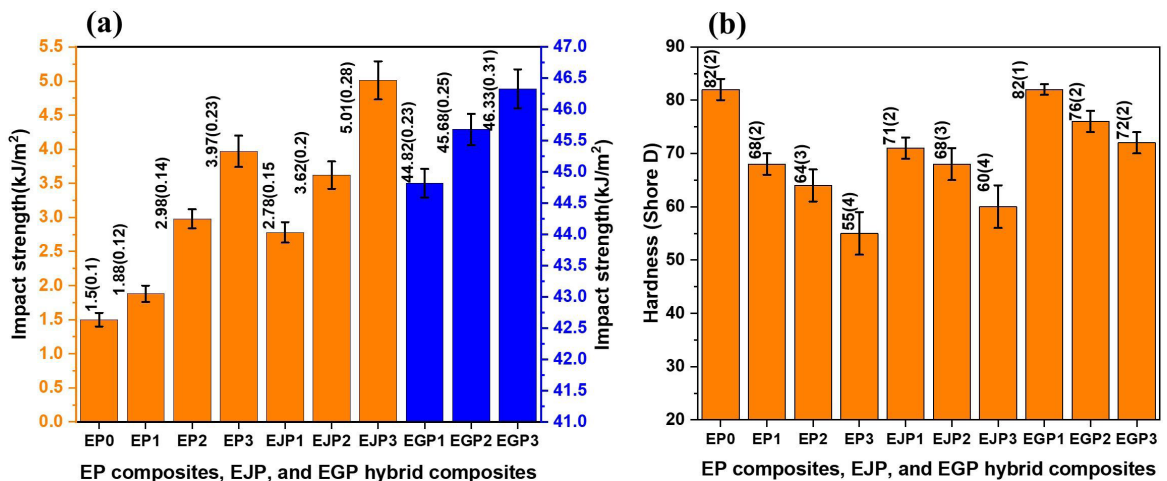


Figure 4. Mechanical characteristics of Epoxy, EP composites, and their hybrid composites: (a) Impact strength, (b) Hardness.

### 3.7. Hardness (Shore D)

The fiber anatomy and distribution of fibers within the matrix determine the hardness of any natural fiber-based composite. Figure 4b depicts the average values and standard deviation of the hardness of each specimen. The incorporation of *PP* stalk particles into the epoxy matrix creates a negative impact on hardness. This could be due to the higher porosity and spongy nature of natural fibers (natural fibers are designed to carry the water and nutrients from one part to another part of the plant). EP1 has a maximum Shore D hardness of 68, which is 17.07% lower than epoxy. Jute and glass fibers have higher modulus and toughness than *PP* stalk particles and cause enhancement in the hardness of the composites. Nearly 4.4% and 20.58% increase in hardness was noticed in

EJP1 and EGP1 hybrid composites, respectively, compared to EP1 composite.

### 3.8. Dynamic Mechanical Analysis (DMA)

The 20 wt.% *PP* stalk particle reinforced epoxy composite (EP2) exhibited excellent mechanical characteristics (tensile, flexural, and compressive strength) compared to its counterparts. And hence only EP2 composite, EJP2 & EGP2 hybrid composites have been considered for the DMA analysis. Figures 5 and 6 illustrate the storage modulus, loss modulus, and tan delta of EP2, EJP2, and EGP2 composites as a function of temperature at various frequencies ranging from 1 to 10 Hz. The storage modulus, loss modulus, and glass transition temperature increased with increasing

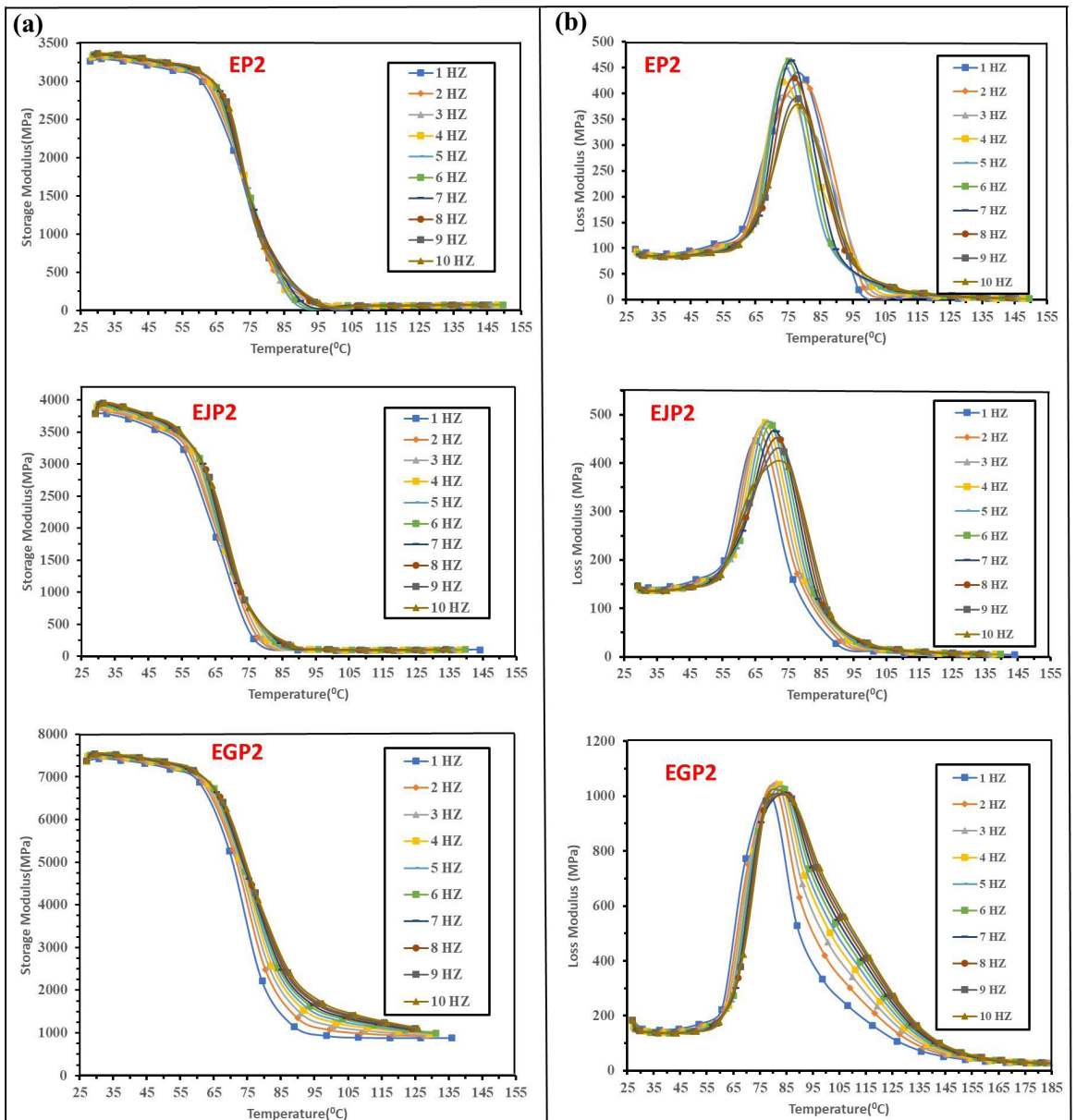
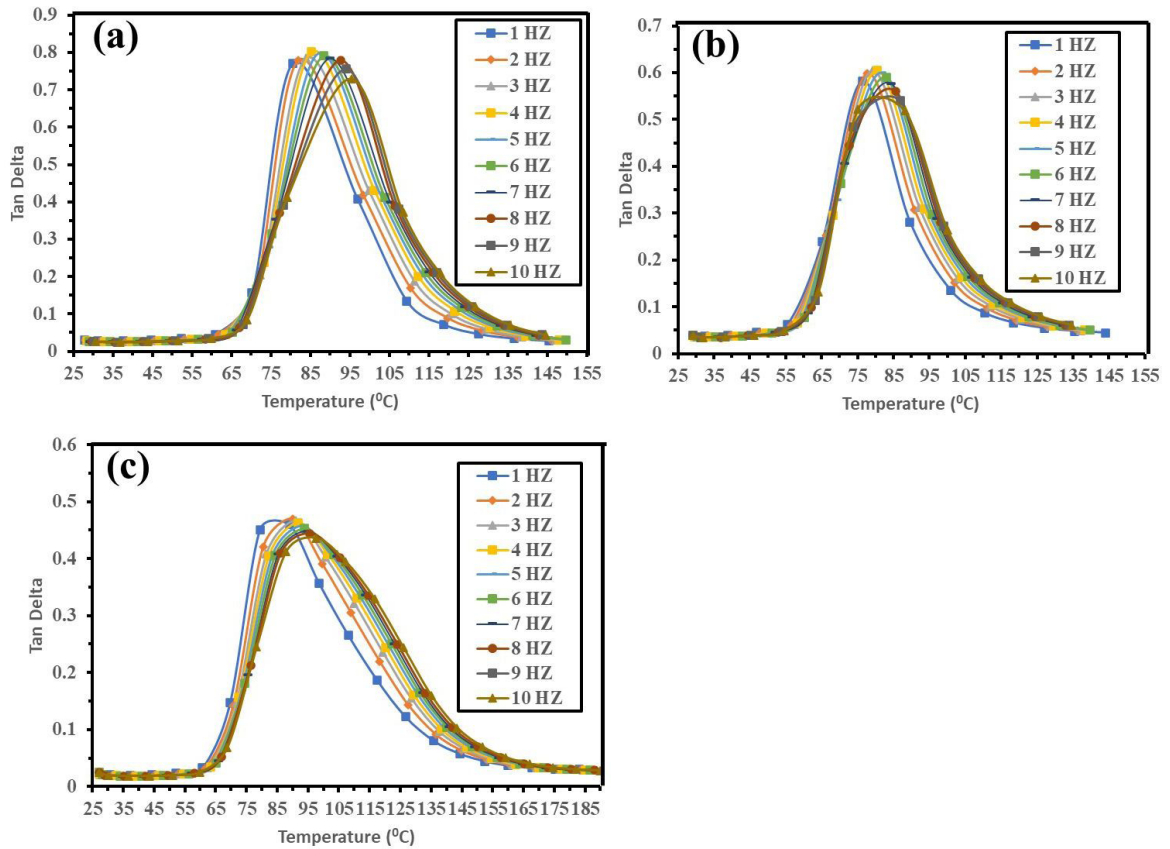


Figure 5. DMA characteristics of EP2 composite, EJP2, and EGP2 hybrid composites: (a) Storage modulus, (b) Loss modulus.



**Figure 6.** Tan delta curves: (a) EP2 composite, (b) EJP2, (c) EGP2 hybrid composites.

frequency in all these composites. This is due to the short period during measurement at higher frequencies. It also shows that the composite stiffness is strongly dependent on the frequency at which it is operated.

The maximum storage modulus observed in EP2 was 3296.1 MPa at a temperature of 30.56 °C (1Hz), which was further enhanced by EJP2 and EGP2 hybrid composites in the order 14.9% and 125.3% at 1Hz, respectively. When the frequency was changed from 1 Hz to 10 Hz, there was a minor change in storage modulus, but a noticeable shift in glass transition temperature and tan delta was observed. The glass transition temperature (from tan delta) of EP2 composite has been shifted from 80.51 °C to 76.43 °C and 89.0 °C in EJP2 and EGP2 hybrid composites, respectively. Glass transition temperature shifting towards higher temperatures may be explained by the decrease in matrix mobility as a result of the presence of jute fibers and glass fibers in hybrid composites. Similarly, the magnitude of the tan delta was decreased by 24.5% and 40.4% in EJP2 and EGP2 hybrid composites compared to the EP2 composite. This demonstrates an effective stress transfer, the reduction of polymer chain mobility, and the enhancement of interlocking of fiber and matrix in the composite. In addition, the degree of (*PP* stalk particles, jute, and glass) fiber adhesion to the matrix controls the mobility of the polymer chain and retards the softening of the composite. This leads to enhancing the dynamic characteristics of these composites. A study of

natural fiber-based hybrid composites (jute/oil palm/epoxy) revealed improved dynamic mechanical properties<sup>26</sup>. Here, the storage modulus varies from 3300 to 3600 MPa, and the tan delta varies from 0.24 to 0.26 based on the jute weight percentage in the hybrid composite. Dynamic mechanical analysis of glass/banana polyester composites<sup>27</sup> has been conducted over a wide temperature range and over three different frequencies, resulting in a decrease in damping factor (approximately 0.2) and an increase in storage modulus (about 8800 MPa) compared to neat polyester. Similar behavior concerning the enhancement of storage modulus and reduction in damping ratio (tan delta) were observed in the investigation of palm/jute hybrid composites<sup>28</sup>, Curaua/epoxy<sup>29</sup>, and Fique fabric/epoxy<sup>30</sup> composites.

### 3.9. Water absorption analysis

Natural fiber-based composites are naturally hydrophilic; hence they must be evaluated for water absorption. A hydroxyl group in *PP* stalk fiber makes it more water-absorbent. In water absorption tests, all *PP* stalk composites and their hybrid composites have attained their maximum water absorption at 25 days and then showed a negligible absorption except for EP3 composite and EJP3 hybrid composite. EP3 and EJP3 composites had a more significant water uptake than other composites. This could be due to an inadequate quantity of epoxy in these composites, resulting in poor wetting of *PP* stalk particles (agglomeration), thereby allowing the water to



diffuse through the composites. In addition, the increased void percentage also contributed to raising the water absorption. Here, the void content increases composites' surface area, promoting more water absorption. EP composites and their hybrid composites (up to 20 wt.% of fiber) revealed an excellent water resistance capability compared to bagasse fiber-reinforced epoxy composites<sup>21</sup> where nearly more than 10% water absorption was noticed. The composite EP2 has maximum water absorption of 5.17% and was further reduced by 3.72% and 2.62% in EJP2 and EGP2 hybrid composites. All composites were stable for up to 30 days except the EP3 composite, where more swelling was noticed.

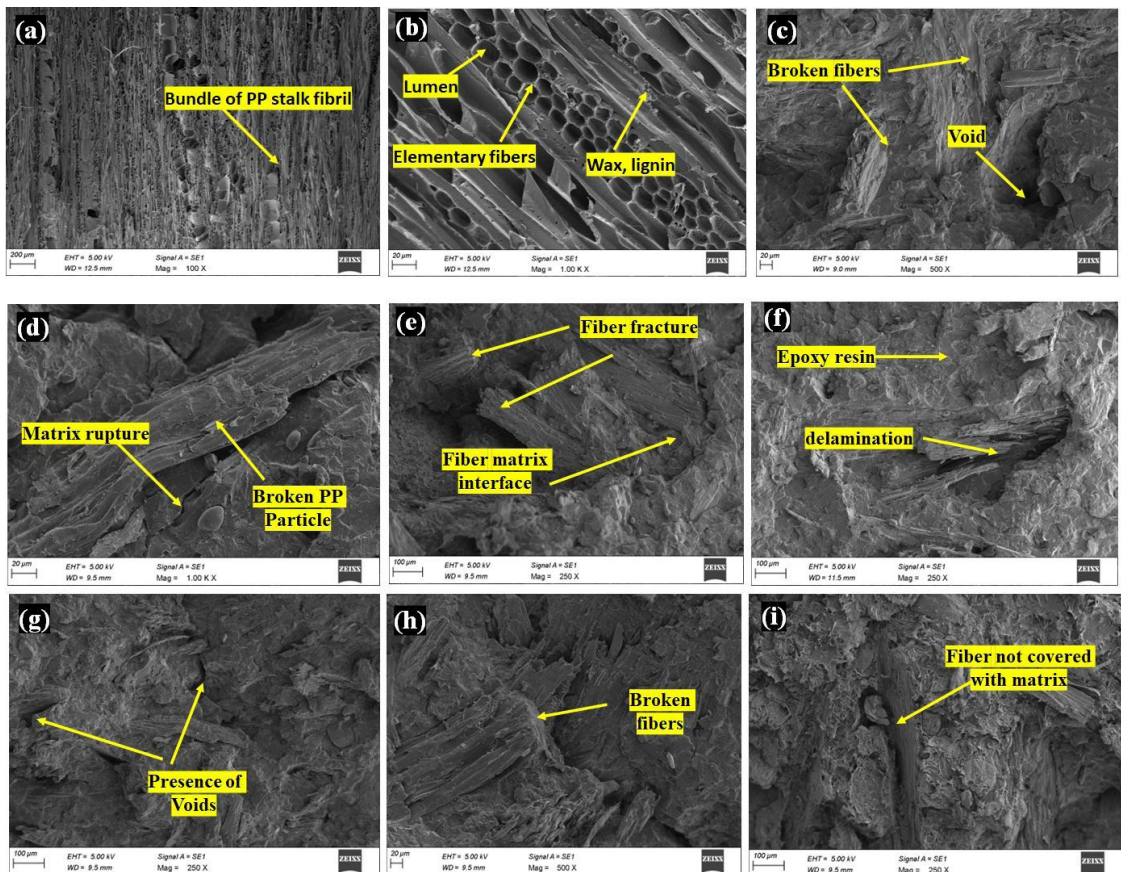
### 3.10. Morphological studies

Morphological studies were carried out on tensile and flexural fractured specimens of EP2 composite to reveal the fiber-matrix interactions. Figures 7a and 7b show bundles of *PP* stalk fibrils with hemicellulose and lignin. The SEM images corroborate the presence of components such as waxes, oils, and extractives, which is evidenced by similar work on the production of xylooligosaccharides<sup>13</sup>. Natural fibers' chemical composition and physical structure are generally complicated and vary from plant to plant. Each fiber is a composite consisting of cellulose microfibrils embedded in hemicellulose and lignin matrix. These cellulose fibers would form hollow structures during helical winding along

with the fiber axis. Figure 7a shows a longitudinal section and (b) shows a transverse section of *PP* stalk particles. Figures 7c, 7d, 7e, and 7f show a tensile fractured surface of the EP2 composite. Micrographs revealed a brittle fracture of epoxy and *PP* stalk particles in the composites. Pull-out of some of the particles were seen in these images due to the lack of adhesion between *PP* stalk particles and matrix in the composite. *PP* stalk particles are pulled out of the matrix when the stress exceeds the fiber/matrix interfacial strength between the filler and the matrix. This debonding or pulling out of the *PP* particles can promote local plastic deformations in the matrix to dissipate fracture energy. The voids, fiber breakage, and matrix rupture were also visible in these images. Figures 7g, 7h, and 7i show the SEM images of flexural fractured specimens. A flexural load causes both the *PP* stalk particle and matrix to fracture, rather than fiber pull-out as in tensile fracture. The fractographic images confirmed the excellent adhesion of *PP* stalk particles to the epoxy matrix. Additionally, these images confirmed that the fibers provide sufficient crack arrest, which leads to better flexural properties.

### 3.11. Ranking of composites using the TOPSIS method

The "technique for order of preference by similarity to ideal solution" (TOPSIS) is a powerful technique for selecting



**Figure 7.** SEM images of *PP* stalk and EP2 composites: (a) and (b) *PP* stalk particles; (c), (d), (e), and (f) Tensile fractured specimens; (g), (h), and (i) Flexural fractured specimens.

the best possible solution from the available alternatives. This technique considers the shortest distance from the best possible solution and the longest distance from the negative-best solution<sup>31</sup>. The main objective of the TOPSIS in this study is to select and order the best (top-ranked) composites based on their tensile strength, flexural strength, impact strength, compressive strength, hardness, water absorption percentage, and void percentage characteristics. In addition, here, the TOPSIS method compares all the developed composites and provides the ranking based on their characterization. Table 4 represents the decision matrix where all decision parameters with their values were added for ranking the composite materials.

Table 5 represents the normalized decision matrix. The value of each cell in the matrix is calculated from Equation 6.

$$r_{ij} = \frac{x_{ij}}{\sqrt{\sum_{i=1}^m (x_{ij})^2}} \quad (6)$$

Here, *i* indicates row, *j* indicates column, and *m* indicates rows number. The weighted normalized decision matrix can be obtained using the Equation 7. To determine the values of this matrix, the weightage for each decision parameter must be given. In this study, the Shannon entropy method is followed to decide the weightage of each parameter. The obtained weights for each parameter are; tensile strength=0.15, flexural strength=0.15, impact strength=0.15, compressive strength=0.15, hardness=0.15, ILSS=0.15, water absorption percentage=0.05, and void percentage=0.05. Table 6 provides separation measure, relative closeness factor, and ranking

of the composite materials. Separation measures ( $S_i^-$ ,  $S_i^+$ ) were calculated using ideal best and ideal worst solutions from the weighted normalized decision matrix. The relative closeness factor ( $c_i$ ) is calculated from Equation 8.

$$v_{ij} = w_j r_{ij} \quad (7)$$

$$c_i = \frac{(S_i^-)}{\left( (S_i^+) + (S_i^-) \right)} \quad (8)$$

The ranking of EP composites, EJP, and EGP hybrid composites was assigned based on the order of relative closeness factor. The hybrid composite EGP2 was assigned rank 1, showing the best properties among all composites as per the TOPSIS method.

### 3.12. Comparison with other natural fiber-reinforced composites

As a result of growing environmental concerns, the utilization of leftover abundant agricultural byproducts (residues) as reinforcement or filler to develop the composites has become increasingly prevalent. Natural fiber-reinforced composites with proper material selection can significantly satisfy the societal requirements and save our future generations from hazardous synthetic materials. A handful of attempts have been documented using agricultural residue-reinforced composites with epoxy and other matrix materials. Natural fiber-reinforced composites can demonstrate a wide range of mechanical characteristics due to various parameters such as fiber architecture, fiber size, fabrication techniques, fiber

**Table 4.** Decision matrix.

Composite specimen	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m <sup>2</sup> )	Compressive Strength (MPa)	Hardness (Shore D)	ILSS (MPa)	water absorption (%)	void (%)
EP1	20.8	33.64	1.88	62.83	68	18.7	4.52	3.9
EP2	24.95	49.3	2.98	71.84	64	16.5	5.17	4.9
EP3	21.87	39.13	3.97	57.99	55	15.45	9.35	7.8
EJP1	21.52	54.38	2.78	65.33	71	22.33	4.32	2.5
EJP2	30.91	59.38	3.62	73.11	68	18.75	4.98	4.2
EJP3	27.45	51.54	5.01	61.69	60	18.3	9.14	6.5
EGP1	48.4	91	44.82	74.15	82	31	3.11	2.4
EGP2	54.04	111.92	45.68	83.12	76	28.75	3.82	3.7
EGP3	48.16	109.89	46.33	73.88	72	25.92	6.11	6.3

**Table 5.** Normalized decision matrix.

Composite specimen	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m <sup>2</sup> )	Compressive Strength (MPa)	Hardness (Shore D)	ILSS (MPa)	water absorption (%)	void (%)
EP1	0.1958	0.1550	0.0237	0.3004	0.3291	0.2786	0.2516	0.2598
EP2	0.2349	0.2271	0.0375	0.3434	0.3097	0.2458	0.2878	0.3264
EP3	0.2059	0.1803	0.0500	0.2772	0.2662	0.2302	0.5200	0.5196
EJP1	0.2026	0.2505	0.0350	0.3123	0.3436	0.3327	0.2403	0.1665
EJP2	0.2910	0.2736	0.0455	0.3495	0.3291	0.2793	0.2771	0.2798
EJP3	0.2580	0.2374	0.0630	0.2949	0.2904	0.2726	0.5084	0.4330
EGP1	0.4556	0.4192	0.5640	0.3545	0.3969	0.4619	0.1728	0.1599
EGP2	0.5087	0.5156	0.5748	0.3974	0.3678	0.4283	0.2122	0.2465
EGP3	0.4533	0.5063	0.5830	0.3532	0.3485	0.3862	0.3398	0.4197

distributions, chemical treatments, fabrication conditions, matrix materials, etc. Table 7 provides the important mechanical properties (tensile strength, flexural strength, and impact strength) of different natural fibers (agro-residues, purposely grown fibers, and wood fibers) reinforced epoxy, polypropylene, polyester, and polylactic acid composites. The developed *PP* stalk composites have comparable mechanical properties with some natural composites (provided in Table 7)<sup>32-46</sup>. Therefore, the *PP* stalk particle reinforced epoxy composites could be a viable alternative

to some natural fiber-reinforced composites and could be used in lightweight structural applications.

#### 4. Conclusions

In this study, it has been demonstrated that *PP* stalk particles can successfully be used as reinforcement to develop EP composites and their hybrid composites. Various characterizations on these composites have led to the following conclusions.

**Table 6.** Separation measure, relative closeness factor, and ranking of composites.

Composite specimen	Ideal separation ( $S_i^+$ )	Negative ideal separation ( $S_i^-$ )	Relative closeness factor ( $c_i$ )	Rank
EP1	0.1152	0.0415	0.2648	7
EP2	0.1079	0.0408	0.2743	6
EP3	0.1163	0.0247	0.1749	9
EJP1	0.1052	0.0486	0.3161	5
EJP2	0.0992	0.0474	0.3230	4
EJP3	0.1049	0.0310	0.2279	8
EGP1	0.0179	0.1158	0.8659	2
EGP2	0.0083	0.1231	0.9370	1
EGP3	0.0231	0.1151	0.8327	3

**Table 7.** Comparative assessment of mechanical properties of PP stalk fiber-reinforced composites with other natural fiber-reinforced composites.

Polymer matrix	Natural fiber	Fiber loading (% w/v)	TS (MPa)	FS (MPa)	IS (kJ/m <sup>2</sup> )	Reference
Epoxy	PP stalk	20	24.95	49.3	2.98	Present work
	Jute/PP hybrid	20	30.91	59.38	3.62	
	Glass/PP hybrid	20	54.04	111.92	45.68	
Epoxy	Bagasse	30	29.23	-	4.5 (J/m)	21
Epoxy	Groundnut shell	12.5	36.66	43.43	-	32
	Rice husk	12.5	12.71	22.72	-	
Epoxy	Banana	16	16.39	57.53	2.25	33
Epoxy	Coir	30	13.05	35.42	17.5	39
Epoxy	Date palm	50	36.17	58.2	10.69 (J/m)	40
	Sunflower husk	15	25	42	-	
	Hazelnut shell	15	28	58	-	41
Epoxy	Walnut shell	15	35	48	-	
	Coconut shell	15	41.3	68.25	-	42
Epoxy	Wood apple	15	43.6	78.19	-	
Epoxy	Soya fiber	20	53.56	94.59	3.61	43
Epoxy	Napier grass	20	28.45	56.21	-	44
Epoxy	Curaua	50	30.29	67.45	-	28
Polyester resin	Ricinus communis	40	20.1	43.2	41.4	45
Unsaturated polyester	Sisal/ silk hybrid	25	18.94	46.18	-	46
Polypropylene	Pineapple/Betel nut hybrid	10	28	44	-	34
Polypropylene	Kenaf	30	15.83	29.34	14.5	35
	Pineapple	30	17.07	45.25	15.0	
Polypropylene	Pigeon pea	30	25.0	47.0	28 (J/m)	16
Polypropylene	Banana peel	20	26.3	38.8	29 (J/m)	36
Polypropylene	Betel nut	30	27.0	55.0	1.6	37
Polylactic acid	Juliflore wood	20	24.89	67.73	1.09	38

Abbreviations: Tensile strength (TS), flexural strength (FS), impact strength (IS).

- EP composites and hybrid composites (EJP and EGP) up to 20 wt.% *PP* stalk particles exhibited less than 5% void content. The EP2 composite had a maximum tensile strength of 24.95 MPa. An enhancement of 23.88% and 147% was observed in EJP2 and EGP2 hybrid composites. The maximum young's modulus noticed in EP composites was 1820 MPa (EP3). This was further enhanced in EJP and EGP hybrid composites by 8.8% and 11.8%, respectively.
- The ILSS was significantly improved in EJP1 and EGP1 composites by 19.3% and 65.77% compared to the EP1 composite. The EP2 composite exhibited a maximum flexural strength of 49.3 MPa. The EJP2 and EGP2 hybrid composites showed nearly 20.4% and 127.0% improvement in flexural strength.
- The EP2 composite, EJP2, and EGP2 hybrid composites exhibited an increase in compressive strength of 6.5%, 8.4%, and 23.25% compared with the epoxy specimen. The maximum impact strength noticed in the EP3 composite was 3.97 kJ/m<sup>2</sup>. A 26.2% increase in impact strength was noticed in the EJP3 hybrid composite compared to the EP3 composite. The impact strength of the EGP3 composite tremendously increased by 11.6 times compared to the EP3 composite. The EP1 composite has shown a maximum Shore D hardness of 68 and nearly 4.4% and 20.58% increase in hardness was noticed in EJP1 and EGP1 hybrid composites, respectively.
- The maximum storage modulus observed in the EP2 composite was 3296.1 MPa, which was further enhanced by EJP2 and EGP2 hybrid composites in the order 14.9% and 125.3% at 1Hz, respectively. *PP* stalk composites and its hybrid composites up to 20 wt.% of particles revealed an excellent water resistance characteristic. SEM images revealed the strong affinity between *PP* stalk particles and epoxy matrix. Micrographs revealed a brittle fracture of epoxy and *PP* stalk particles in the composites. The hybrid composite EGP2 was assigned rank 1, showing the best properties among all composites as per the TOPSIS method.

Compared to other natural fiber-based composites, *PP* stalk particle reinforced epoxy composites and their hybrid composites (up to 20 weight percentage of *PP* stalk particles) have demonstrated comparable mechanical properties. Thus, this new class of composites could be employed as a viable alternative to some natural fiber-reinforced composites and could be used in lightweight structural applications such as automobile interior parts, furniture construction, indoor civil constructions, packaging containers, etc.

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